JRC Scientific and Technical Reports

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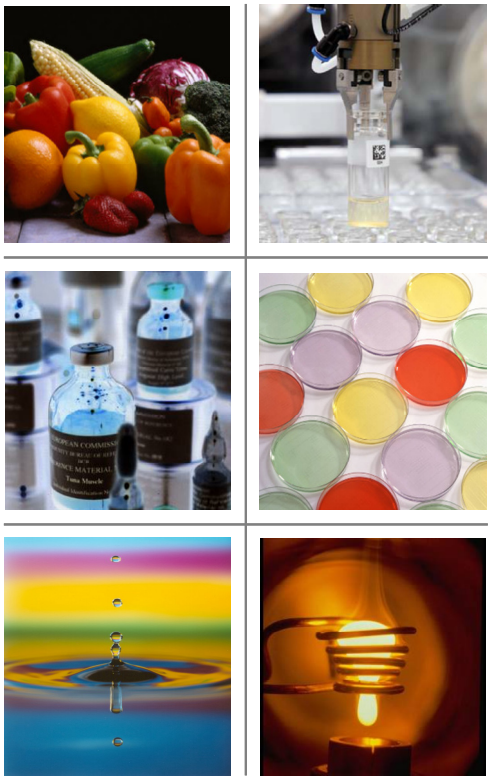
A. Verbruggen, J. Bauwens, R. Eykens, U. Jacobsson, R. Jakopic, F. Kehoe, H. Kühn, Y. Kushigeta, S. Richter, Y. Aregbe



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Institute for Reference Materials and Measurements

The mission of the JRC-IRMM is to promote a common and reliable European measurement system in support of EU policies.

European Commission
Joint Research Centre
Institute for Reference Materials and Measurements

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IRMM information
REFERENCE MATERIALS

Preparation and certification of IRMM-1027m, Large-Sized Dried (LSD) spike

IRMM-1027m

A. Verbruggen, J. Bauwens, R. Eykens, U. Jacobsson, R. Jakopic, F. Kehoe, H. Kühn, Y. Kushigeta, S. Richter, Y. Aregbe

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Disclaimer

Certain commercial equipment, instruments, and materials are identified in this report to specify adequately the experimental procedure. In no case does such identification imply recommendation or endorsement by the European Commission, nor does it imply that the material or equipment is necessarily the best available for the purpose.

Contents

Summary

Large sized dried spikes (LSD) have become a fundamental part of the fissile material control of irradiated nuclear fuel. In the frame of providing these spikes to the nuclear industry, a new set of LSD Spikes for the determination of uranium and plutonium by isotope dilution mass spectrometry in solutions of spent fuel from reprocessing plants has been prepared and certified for uranium and plutonium isotopic contents. The methodology followed was comparable to that of previous batches. The solution, made by dissolution of the starting materials in nitric acid, was dispensed directly into individual penicillin vials. As in previous campaign an automated system was introduced to dispense and weigh the vials.

The new batch of large size dried spikes contains ca. 50 mg of uranium with ^{235}U amount fraction of 19.5% and ca. 1.8 mg of plutonium with ^{239}Pu amount fraction of 97.8% in each individual vial, covered with a light layer of organic material (cellulose acetate butyrate) as stabilizer.

The U and Pu amount content was certified based on values from mass metrology of the validated automated system. Verification of the amount contents of the spike was done by IDMS at IRMM. The values measured for the dried covered spikes agreed well with those calculated from the weights of starting materials dissolved and the weights of the final solution.

Introduction

The series IRMM-1027m Large Size Dried (LSD) Spikes is being prepared to fulfil the existing requirement for reliable and traceable spikes in fissile material control of dissolved nuclear fuel. The amount content of the spikes is such that no dilution of a typical sample of dissolved fuel is needed before measurement by Isotope Dilution Mass Spectrometry (IDMS) using a single LSD spike. Because each spike is certified for amounts of plutonium and uranium in the vial, the only quantitative step needed at the reprocessing plant laboratory is to weigh as accurately as possible an aliquot of the dissolved fuel solution onto the spike and ensure complete mixing of spike and sample.

The plutonium component is highly enriched in ^{239}Pu and is used to measure the Pu content in the fuel. Approximately 1.8 mg Pu is contained in each LSD spike. The uranium component is a mixture of two uranium source materials, natural uranium and a highly enriched uranium component. These materials are mixed to arrive at a final enrichment of just under 20% relative amount fraction of ^{235}U, which means for accountability purposes the uranium is classified as ‘low enriched’.

High purity metals are chosen as starting materials. It was decided to use CETAMA MP2 plutonium metal and uranium metals EC NRM 101, CRM-116 as in most previous batches. This allows the isotopic contents of the LSD spike to be certified from the certificates of the metals (chemical purity and isotopic content), the weights of the metals and the solution. As a result the values of the uranium and plutonium isotopic contents of the final certified spike solution have low uncertainties which are directly traceable to the SI via the masses of the starting materials.

A single large volume of batch solution is made up; 1200 units were dispensed by the automated system, into penicillin vials. The solution in each vial is dried down and then covered with a light organic coating dried onto the spike material. The coating (cellulose acetate butyrate, CAB) was also used for previous batches. It provides a fixed layer to hold the dried spike material on the base of the vial dissolves quickly in warm nitric acid and has no significant effect on the subsequent IDMS measurements.

Following the experience of previous series, the isotope amount contents of a set of individual spike vials after drying are measured by isotope dilution to verify the values from the mass-metrology of the starting metals dissolved and the weight of the final solution.

A similar verification procedure is applied on the batch solution but due to technical reasons this was not accomplished for the IRMM-1027m batch.

Dissolution of standard materials

Pu metal Cetama MP2

The metal standard is delivered in a flame-sealed vial with a certified mass of Pu metal. Four ampoules of MP2 were required for the preparation of this LSD spike. Each vial was cut open, the Pu removed with tweezers, weighed and placed in the 3 L borosilicate flask (see next paragraph). The total amount of Pu, calculated to obtain a solution of ca. 0.7 mg plutonium per gram solution when dissolved in 3 kg nitric acid, was weighed at IRMM. The results from the weighing of metal agreed well with the CETAMA certified mass of the MP2 metal.

Uranium metals EC NRM 101, CRM-116

Approximately 47.8 g EC NRM 101 (natural uranium) metal was etched with 1 M HNO_3 as recommended on the certificate to remove surface oxides, rinsed with de-ionised water then acetone and finally dried. The metal was accurately weighed and added to the flask containing the Pu solution. The same was done with 12.1 g NBL CRM-116 enriched uranium. The masses of the uranium were calculated so as to yield a solution of ca. 20 mg uranium per gram solution with an enrichment of ca. 19.5% relative amount fraction of ^{235}U.

Making up the batch solution

The dissolution was carried out entirely in a 3 L long-necked borosilicate flask that had been cleaned in the IRMM MCL (Medium-Clean Chemistry Laboratory). All weighings were carried out as accurately as possible, with reference to a set of calibrated weights traceable to the international kilogram prototype at BIPM, Sèvres. The necessary corrections for air buoyancy effects, taking into account the ambient pressure, temperature, humidity and the density of the material were made.

The weighed Pu metal was transferred into the flask. Concentrated nitric acid and a few drops of conc. HF were added and the flask was warmed to about 90° C to dissolve the Pu. The dissolution was controlled visually and took several weeks to be complete. After cooling the solution was kept under controlled conditions to ensure complete Pu dissolution before the uranium was added. The uranium dissolved completely within a few days.

The complete dissolution of the metals and the solution homogeneity was ensured by allowing the solution to stand for at least 8 weeks after the starting materials had been adjudged to be completely dissolved.

After making up the solution to the prescribed mass of 3.0 kg, the solution was left for another 4 weeks to homogenise with occasional swirling by hand.

Measurement of isotopic abundances in the batch solution

The verification of the certified ratios for uranium was accomplished by mass spectrometry measurements on aliquots of the batch solution. A plastic syringe was filled from the batch solution and from this syringe an aliquot of 1 g of solution was processed for measurement of isotopic ratios.

The chemical procedure prior to mass spectrometry as detailed in [1] was employed. A 1 M HNO_3 solution of uranium and of plutonium separated was prepared for measurements of the isotopic ratios by TIMS.

The isotopic ratios of the uranium were measured on the Finnigan Triton [2] , following IRMM quality management procedures for uranium. The mass spectrometers were calibrated for mass fractionation by measuring IRMM-074/10 uranium isotopic reference material during the procedure. A turret was loaded with 6 filaments of IRMM-1027m and 6 filaments of IRMM-074/10.

The measured ratios compared to the calculated values from the certificates are listed in Table 1 for uranium. The measured ratios for uranium are taken from the Triton

measurements and are compared to the ratios calculated from the mixing of the two metals and their certified isotopic abundances.

The verification of the certified ratios for plutonium was deemed not to be required due to the use of certified primary plutonium reference material, MP2 metal, and the fact that no mixing of contamination was expected from other materials used in the preparation as proven in previous preparations of LSD spikes.

Table 1: Isotopic amount ratios of uranium in the batch solution. Values from certificates and metrological weighing are compared with abundances calculated from measurement of isotopic ratios in a sample of the batch solution. Expanded uncertainties are given in brackets (coverage factor k=2).

	$n(^{234}U)/n(^{238}U)$	$n(^{235}U)/n(^{238}U)$	$n(^{236}U)/n(^{238}U)$
Certified value	0.002 584(10)	0.243 893(56)	0.001 063 1(32)
Measured value	0.002 586 0(23)	0.243 941(47)	0.001 065 8(17)

Aliquoting of batch solution

The solution in the flask was re-weighed and adjusted for the small evaporation losses during the time the verification of the batch measurements were done. Prior to dispensing, the vials were cleaned following working instruction "Cleaning of glass penicillin vials for storage of LSD spikes", pre-engraved with the reference material name (IRMM-1027m) and an individual running number starting at 0001.

Automated system aliquoting

The automated system to produce LSD spikes has been installed in collaboration with Nucomat, a company with a recognized reputation in design and development of integrated automated systems. The major components of the system are a robot, two balances, a dispenser and a drying unit fitted into a glove box [3].

The robot is software driven and designed to control all movements inside the glove-box, to identify the penicillin vials with a barcode reader, to dispense the LSD batch solution into the vials and to weigh the amount dispensed. The weighing section is equipped with a semi-analytical balance (Sartorius TE124S) and a 5 kg balance (Sartorius TE6101) to monitor the mass of the mother solution during dispensing and to verify overnight losses by evaporation.

The LSD spike solution was weighed into the vials over a period of 5 days following working instruction 'LSD automated system equipment manual'). Batches of 48 vials were prepared and kept in a perspex holder that fitted into a plastic box and each box was closed and stacked with the others ready for drying. The boxes with the penicillin vials were transferred into one of the drying glove-boxes for the next processes: drying and covering with CAB.

Drying solutions and addition and drying of CAB

The solutions were treated according to the procedure "Processing & Preparation of Large-Size Dried (LSD) Spikes following dispensing" which includes drying by gentle heating on a thermostatically controlled hot-plate at approx. 60 °C. When the solutions had dried (typically 4-5 days continuous heating), about 0.7 mL of a 10% cellulose acetate butyrate (CAB) solution in acetone was added, the solution allowed to evaporate at room temperature for 3 hours and then heated at approx. 45 °C for up to 45 min to dry completely.

Two separate glove-boxes were used for the drying allowing up to 48 samples per week to be dried and covered with CAB. The vials containing dried samples were stacked horizontally and inspected regularly. If the material appeared to have flowed even slightly in the vial the vial was heated again to remove the last traces of solvent. The vials containing the dried material covered with CAB were closed with an iso-versilic stopper and an aluminium cap. The vials were then labelled and sealed in PVC packages for storage.

Drying, coating with CAB layer and packing were carried out over a period of several months.

Verification of Pu amount content in selected vials

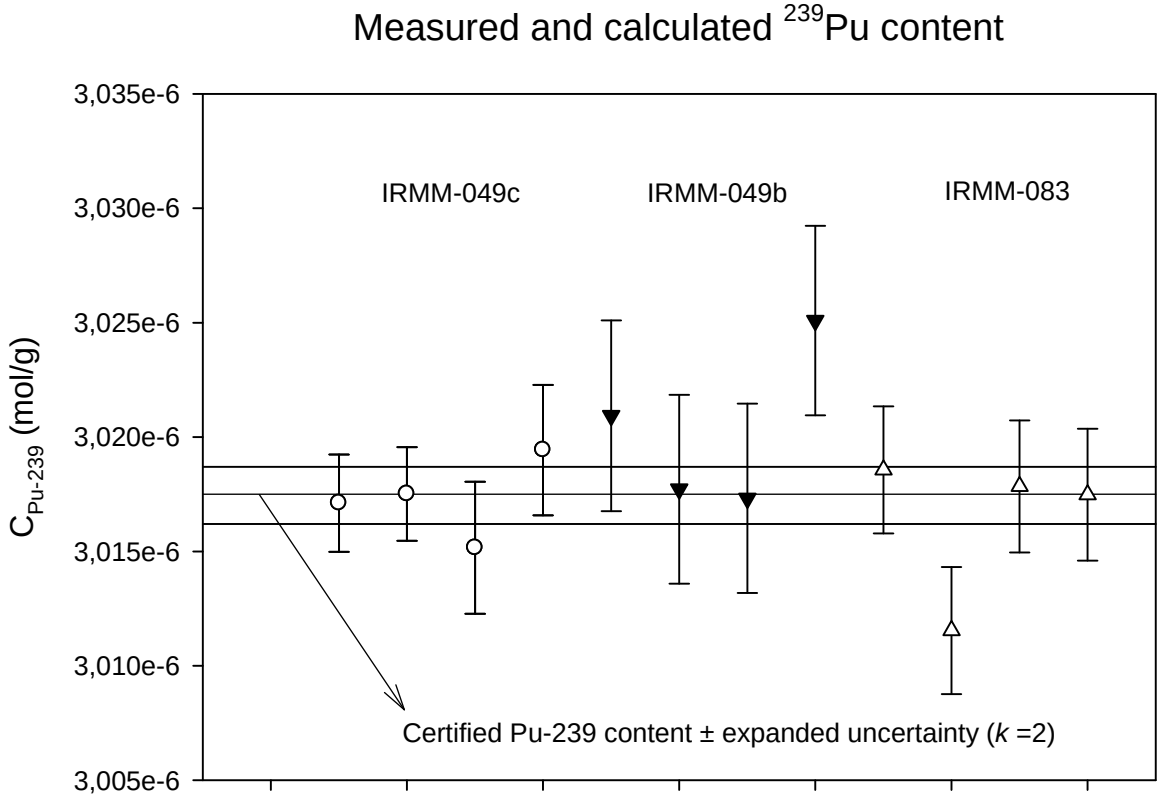
After drying and CAB covering were complete, three series of four vials were randomly stratified chosen for verification measurements on Pu. To each of the series of four respectively, for the first series 5 g of IRMM-049c ^{242}Pu spike, for the second series 2.5 g of IRMM-049b ^{242}Pu spike and for the third series 0.5 g IRMM-083 ^{240}Pu spike were weighed in and the standard IDMS procedure and the working instructions above were used for the measurement of Pu amount content in the spikes. The plutonium ratios $n(^{239}\text{Pu})/n(^{242}\text{Pu})$ and $n(^{239}\text{Pu})/n(^{240}\text{Pu})$ for IRMM-083 were measured on the Pu-Triton.

The results of the verification measurements described above are given in Table 2, with mean values of the series and shown in Figure 1. These measurements gave mean values that agreed well with the values for plutonium amount content calculated from the amounts of dissolved metal and solution.

Table 2: Amount content of ^{239}Pu in mol·g^{-1}. Values from the certificate and metrological weighing are compared with mean values calculated from measurement of individual vials. Expanded uncertainties are given in brackets (coverage factor k=2).

Certificate	Vials series 049c	Vials series 049b	Vials series 083
3.017 5(12) · 10^{-6}	3.017 3(32) · 10^{-6}	3.020 3(38) · 10^{-6}	3.016 4(33) · 10^{-6}

Figure 1: 'Metrological' concentration of ^{239}Pu in IRMM-1027m (from the weights of metal and solution) compared with the measured values by IDMS (with expanded uncertainties coverage factor k=2).



Verification of U amount content in selected vials

For uranium a set of six vials were randomly stratified chosen for verification measurements. To each of these, 5 g of IRMM-046b spike was weighed in, the standard IDMS procedure and the working instructions above were used for the measurement of U amount content in

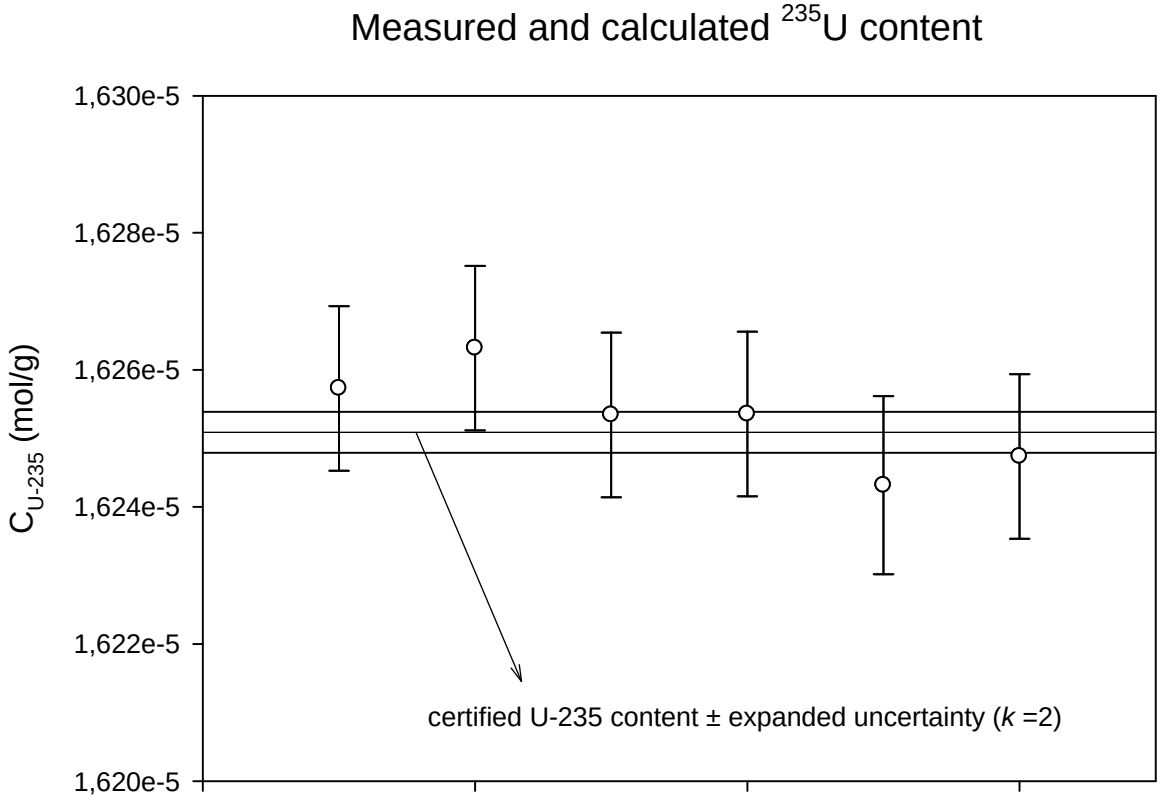
the spikes. The uranium isotopic ratios $n(^{235}U)/n(^{233}U)$ were measured on the Triton. As IRMM-046b is in the process of being recertified, a tentative preliminary ^{233}U amount content was used in the calculations.

The results of the verification measurements described above are given in Table 3 and shown in Fig. 2. These measurements gave values that agreed well with the values for uranium amount content calculated from the amounts of dissolved metals and solution.

Table 3: Amount content of ^{235}U in $mol \cdot g^{-1}$. Values from the certificate and metrological weighing are compared with mean values calculated from measurement of individual vials. Expanded uncertainties are given in brackets (coverage factor k=2).

Certificate	Vials series 046b
$1.625\ 09(30) \cdot 10^{-5}$	$1.625\ 26(58) \cdot 10^{-5}$

Figure 2: ‘‘Metrological’ concentration of ^{235}U in IRMM-1027m (from the weights of metals and solution) compared with the measured values by IDMS (with expanded uncertainties coverage factor k=2)..



Conclusion

A new series of LSD spikes for IDMS determinations of uranium and plutonium contents in solutions of spent nuclear fuel from reprocessing plants has been prepared.

The certification of the spike is based on the metrological data, the certificate of the base materials and the verification measurements. The final certification values are established by mass-metrology of the metals and the solutions.

The verification of the certified values from the mass-metrology was accomplished by IDMS measurements on the batch solution and individual vials. The agreement was satisfactory.

The materials prepared are commercially available from IRMM, Geel as reference material IRMM-1027m for application in the nuclear safeguards measurements of uranium and plutonium in input solutions.

References

[1] Preparation and Certification of a new Type of Large Size Dried Spikes, Batch IRMM-1027f, A Alonso, R Eykens, F Kehoe, H Kühn, N Surugaya, A Verbruggen, R. Wellum, GE/R/IM/36/02

[2] New Procedures for Uranium Isotope Ratio Measurements using the new TRITON Thermal Ionisation Mass Spectrometer, S. Richter, A. Alonso, H. Kühn, R. Wellum, P.D.P. Taylor, Report EUR 21849

[3] An automated system for the preparation of Large Sized Dried (LSD) Spikes, A. Verbruggen, J. Bauwens, N. Van De Steene, U. Jakobsson, R. Eykens, R. Wellum, Y. Aregbe, ATALANTE Conference 2008, Montpellier (France), May 19-22, 2008

Annex 1: Certificate of uranium metal: EC NRM-101

Certified Nuclear Reference Material

Certificate of Analysis

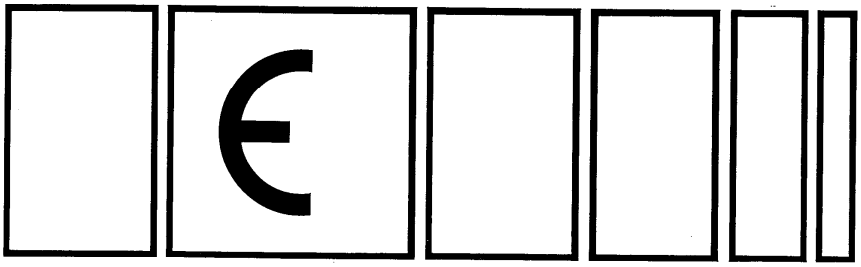
EC NUCLEAR REFERENCE MATERIAL NO. 101

MATERIAL : URANIUM METAL

URANIUM MASS FRACTION : (999.85 ± 0.05) g·kg-1

The uncertainty has been calculated by multiplying the estimated overall standard deviation by a factor of two. This corresponds to a confidence level of about 95 percent.

Commission of the European Communities
Joint Research Centre
Geel Establishment (CBNM)



Annex 2: Certificate of uranium metal: NBL CRM-116

U. S. Department of Energy
New Brunswick Laboratory

New Brunswick Laboratory
Certified Reference Materials
Certificate of Analysis

CRM 116

Uranium (Enriched) Metal
(Uranium and Uranium-235 Standard)

Uranium (etched metal basis) ..... $99.967_2 \pm 0.006_9$ Wt.%
($\alpha = 0.05$, n = 6)

Uranium-235 ..... $93.121_5 \pm 0.004_7$ Wt.%
($\alpha = 0.05$, n = 6)
$93.183_7 \pm 0.004_7$ At.%

Relative atomic weight 235.201

Metal must be etched in 1 + 1 HNO_3, rinsed in distilled-deionized water and acetone, and dried prior to use.

REFERENCE METHODS OF ANALYSIS: Titrimetry (high precision NBL method) verified with NBL CRM 112-A Uranium Metal Standard and thermal ionization mass spectrometry verified with NBL CRM U930 Uranium Isotopic Standard.

June 1978
Argonne, Illinois

Carleton D. Bingham
Director

Annex 3: Certificate of plutonium metal: Cetama MP2

CETAMA

COMMISSARIAT A L'ENERGIE ATOMIQUE

COMMISSION D'ETABLISSEMENT DES METHODES D'ANALYSE

cea

REFERENCE MATERIAL CERTIFICATE

PLUTONIUM METAL

"MP2"

Sample n° Axxx Mass : 0.xxxxxx ± 0.000012 g
(For the values x see page 4)

The reference material to which this certificate relates is intended for the calibration of chemical composition measurement. The overall chemical content of plutonium is certified. The confidence interval associated with the certified value for a single sample, takes into account uncertainties associated to with analysis and heteregenity of metal. This content, expressed as a percentage of mass, was the following on 12 march 2002 for a single sample with a probability level of 0.95.

99.90 ± 0.04 %

THE TRUE MASS OF THE SAMPLE A ± **12 µg**, RELATED TO A VACUUM, IS THAT INDICATED IN THIS CERTIFICATE AND ON THE AMPOULE.

The possibility of surface oxidation makes it impossible to envisage weighing at the time of use

Isotopique composition is certified on 12 march 2001 : see certificate IRMM page3

The preparation, analysis and certification of the plutonium to which this certificate relates was carried out by different units of the CEA group under the supervision of the Committee for Establishing Analysis Methods (CETAMA).

Le responsable MR

CETAMA
CEA VALRHO Marcoule
B. P. 17171
30207 BAGNOLS SUR CEZE CEDEX FRANCE
Téléphone (33) 4.66.79.69.88 - Télécopie (33) 4.66.79.69.89

CETAMA certifié ISO 9001:2000 par

Version : 06/2001

On 12/03/200, the metal contained around:

- by weight, 489 $mg.kg^{-1}$ of uranium,
- by weight, 438 $mg.kg^{-1}$ of américium..

UTILISATION

The sample, which consists of a piece of metal, is supplied in a double glass ampoule filled with pure nitrogen at a pressure of around 0.1 Pascal.

The ampoule must be opened with care inside a glove box. All the sample must be transferred to the dissolver.

Cover with 0.1 $mol.l^{-1}$ hydrochloric acid. The ampoule must be thoroughly washed with the same acid to recover any particles of metal which may have become separated. In 2 ml fractions, add the necessary quantity of 12 $mol.l^{-1}$ hydrochloric acid of guaranteed purity to obtain a 4 $mol.l^{-1}$ hydrochloric acid solution. Allow dissolving to proceed without heating for 10 to 15 minutes, then heat to boiling point. If there are still particles of plutonium at the bottom of the dissolver after heating for two hours, add 2 ml of 12 $mol.l^{-1}$ hydrochloric acid and 2 drops of 1 $mol.l^{-1}$ hydrofluoric acid and continue heating for another two hours. Repeat the operation if necessary until the material is totally dissolved.
If plutonium fluoride precipitates out, add a few drops of aluminium nitrate (approximately one $mol.l^{-1}$)..
Allow to cool and adjust to the required volume.

ADDITIONAL INFORMATION

The certified plutonium content has been deduced from analysis of impurities carried out by five laboratories and checked by chemical assay of the plutonium in two different laboratories using three different methods of analysis.
Spark Source Mass Spectrometry has given a full analysis of the impurities and, where concentration levels allowed, inductively-coupled plasma atomic emission spectrometry has been used to establish the concentrations of some of them.
The uranium was determined by laser spectrofluorimetry and the americium by gamma spectrometry.Carbon was determined by coulometry, after transformation into gaseous form by combustion in oxygen.
The gases were analysed by chromatography in the aqueous phase:

- for nitrogen and oxygen after extraction by high temperature stream under an inert gas,
- for hydrogen after diffusion in a vacuum.

Annex 4: Certificate of plutonium metal: isotopic abundances IRMM

EUROPEAN COMMISSION
DIRECTORATE GENERAL JRC
JOINT RESEARCH CENTRE
IRMM
Institute for Reference Materials and Measurements

CERTIFICATE of a reference measurement

IM/MeaC/07/116

11 April 2007

SUBJECT : Recertification of CEA CETAMA MP2

1. Applicant: A. Verbruggen

2. Sample Identification:
   - CEA/CETAMA/MP2
   - Chemical form: Pu metal provided by CEA/CETAMA

3. Measurands:
   - Isotopic composition

isotope amount ratio(s)	
$n(^{238}Pu)/\ n(^{239}Pu)$	0.000 030 83(29)
$n(^{240}Pu)/\ n(^{239}Pu)$	0.022 432 4(51)
$n(^{241}Pu)/\ n(^{239}Pu)$	0.000 237 8(31)
$n(^{242}Pu)/\ n(^{239}Pu)$	0.000 075 70(78)

amount fraction (·100)		mass fraction (·100)	
$n(^{238}Pu)/n(Pu)$	0.003 015(29)	$m(^{238}Pu)/m(Pu)$	0.003 002(28)
$n(^{239}Pu)/n(Pu)$	97.773 05(58)	$m(^{239}Pu)/m(Pu)$	97.763 80(59)
$n(^{240}Pu)/n(Pu)$	2.193 28(49)	$m(^{240}Pu)/m(Pu)$	2.202 27(49)
$n(^{241}Pu)/n(Pu)$	0.023 25(30)	$m(^{241}Pu)/m(Pu)$	0.023 44(31)
$n(^{242}Pu)/n(Pu)$	0.007 402(76)	$m(^{242}Pu)/m(Pu)$	0.007 494(77)

molar mass: 239.074 790 8(91) $g \cdot mol^{-1}$

4. Date of sample receipt : n.a.
   Date of completion of measurement : 7 November 2006

5. All uncertainties indicated are expanded uncertainties $U = k \cdot u_c$ where u_c is the combined standard uncertainty estimated following the ISO/BIPM guide[1]. They are given in parentheses and include a coverage factor k=2. They apply to the last two digits of the value. The values certified are traceable to the SI. The primary certified values are the isotope amount ratio ; other values are derived from them. Reproducing the derived values may result in difference due to rounding errors.

[1] International Organisation for Standardisation, Guide to the expression of Uncertainty in Measurement, ©ISO, ISBN 92-67-10188-9, Geneva, Switserland, 1993

Uncertainty budget :

Quantity	Value	Standard Uncertainty	Index
Atomic mass ^{239}Pu	239.05215760 g/mol	$5.1 \cdot 10^{-6}$ g/mol	59.6 %
Measurement ratio 240/239	0.02243535 mol/mol	$3.81 \cdot 10^{-6}$ mol/mol	14.9 %
Measurement ratio 241/239	$240 \cdot 10^{-6}$ mol/mol	$450 \cdot 10^{-9}$ mol/mol	0.9 %
Measurement ratio 242/239	$75 \cdot 10^{-6}$ mol/mol	$175 \cdot 10^{-9}$ mol/mol	0.4 %
variability$_{241/239}$	0.0 mol/mol	$2.65 \cdot 10^{-6}$ mol/mol	21.0 %
variability$_{242/239}$	0.0 mol/mol	$650 \cdot 10^{-9}$ mol/mol	3.0 %
M_{Pu}	239.07478500 g/mol	$6.46 \cdot 10^{-6}$ g/mol	

6. The traceability to SI is established through standards from IRMM-290.

7. Analytical measurement procedure
   - Mass spectrometric measurments were performed by H Kühn an F Kehoe for the $[n(^{238}Pu)/n(^{239}Pu)]$, $[n(^{240}Pu)/n(^{239}Pu)]$, $[n(^{241}Pu)/n(^{239}Pu)]$ and $[n(^{242}Pu)/n(^{239}Pu)]$ using the MAT262 TIMS, sample solutions were prepared for TIMS analysis by F Kehoe. A. Verbruggen was responsible for preparation and issuance of the certificate.
   - The atomic masses, used in the calculation are from G. Audi and A.H. Wapstra.[2]
   - Reference numbers of the measurement data: measurements number T26629, T26A03, T26B07, logged in S:\D04-IM\Secure Data\Project Data\MP2 (based on 081a and LSD1027i)\MP2 IA Summary MAT262 measurements.
   - Full details of the preparation and the certification procedure can be found in certification report EUR*****.

8. These samples will be stored for a minimum period of six months from the date of this certificate

André Verbruggen
Group leader Nuclear Chemistry

Stephan Richter
Group leader Nuclear Mass Spectrometry

Copies
P Taylor, IM unit head
Y Aregbe, Action leader Nuclear Safeguards
F Kehoe
H Kühn

[2] G. Audi and A.H. Wapstra, The 2003 atomic mass evaluation, Nucl Phys A729 (2003) 337-676

Annex 5: Mass Metrology certificate: base materials

EUROPEAN COMMISSION DIRECTORATE GENERAL Joint Research Centre	**Certificate of weighing**	irmm Institute for Reference Materials and Measurements

E. 3701 **Issued date**: 09 December 2008 **Page** 1 of 1

Applicant: Verbruggen **Group:** IM-Nuclear

Project: IRMM-1027 M LSD **IM-unit ref.:**

Description: Preparation mother solution IRMM-1027 M

Date of receipt of request: 21.12.2007 **Weighing date**: 21 May 2008

The reported results applies only to the objects / samples described in this certificate

	Weight in g
Mass of Pu metal (MP 2 BC 2701)	2.2396 (2)
Mass of U metal (NBL-CRM-116)	12.074 (1)
Mass of U metal (EC-NRM-101)	47.771 (4)
Mass of IRMM-1027 M	3031.36 (5)

Observations:

The measurements and uncertainty estimates, were performed according to working instruction WI-0185, "Mass determination by substitution weighing"on balances AT 261 and At 201 with IRMM inventory No 1999 00337 27 and 1996 00547 73.

Traceability:

The certified mass values are traceable to the International Kilogram Prototype via regular calibrations of the IRMM principal kilogram. The sets of working mass standards M 3 and M 10 were used as reference in the mass determination.

Uncertainty:

All reported uncertainties are expanded uncertainties $U = k \cdot u_c$ where u_c is the combined standard uncertainty calculated according to the ISO/BIPM Guide to the expression of Uncertainty in Measurement. The coverage factor $k = 2$ corresponds to a coverage probability of about 95%. U applies to the last digit of the value of the measurement result and is given in parentheses ().

Annexes:

Signature
Mass Metrology Service

Retieseweg, B-2440 Geel, Belgium; Tel.: +32-(0)14-571 211 • Fax: +32-(0)14-571 978• http://www.irmm.jrc.be

Annex 6: Certificate of IRMM-1027m

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for Reference Materials and Measurements
Reference Materials

CERTIFICATE
SPIKE ISOTOPIC REFERENCE MATERIAL IRMM-1027m

This Spike Isotopic Reference Material consists of approximately 2.5 g of solution subsequently evaporated to dryness and covered with a dry layer of circa 50 mg cellulose acetate butyrate (CAB) to ensure spike integrity.

Each unit is identified by a vial number. The sample certified mass of the solution for each vial is listed in table 1.

The Isotopic Reference Material (IRM) is supplied with isotope amount content of ^{235}U, ^{238}U and ^{239}Pu certified to be

1.625 09(30) 10^{-5} mol (^{235}U) · g^{-1} (solution)
6.663 12(75) 10^{-5} mol (^{238}U) · g^{-1} (solution)
3.017 5(12) 10^{-6} mol (^{239}Pu) · g^{-1} (solution)

Other uranium and plutonium isotopes present are related to the ^{238}U and ^{239}Pu concentration through the following certified amount ratios:

$n(^{234}U)/n(^{238}U)$:	0.002 584(10)
$n(^{235}U)/n(^{238}U)$:	0.243 893(56)
$n(^{236}U)/n(^{238}U)$:	0.001 063 1(32)

$n(^{238}Pu)/n(^{239}Pu)$:	0.000 030 22(28)
$n(^{240}Pu)/n(^{239}Pu)$:	0.022 428 0(51)
$n(^{241}Pu)/n(^{239}Pu)$:	0.000 210 2(28)
$n(^{242}Pu)/n(^{239}Pu)$:	0.000 075 71(78)

Retieseweg 111, B-2440 Geel - Belgium. Telephone: (32-14) 571 211. http://irmm.jrc.ec.europa.eu 10/2009

This corresponds to isotopic compositions of uranium and plutonium with the following abundances:

	amount fraction (·100)		mass fraction (·100)
$n(^{234}U)/n(U)$	0.207 13(84)	$m(^{234}U)/m(U)$	0.204 15(83)
$n(^{235}U)/n(U)$	19.549 9(37)	$m(^{235}U)/m(U)$	19.351 6(37)
$n(^{236}U)/n(U)$	0.085 21(26)	$m(^{236}U)/m(U)$	0.084 71(26)
$n(^{238}U)/n(U)$	80.157 8(33)	$m(^{238}U)/m(U)$	80.359 6(33)

	amount fraction (·100)		mass fraction (·100)
$n(^{238}Pu)/n(Pu)$	0.002 954(28)	$m(^{238}Pu)/m(Pu)$	0.002 942(28)
$n(^{239}Pu)/n(Pu)$	97.776 17(56)	$m(^{239}Pu)/m(Pu)$	97.766 94(56)
$n(^{240}Pu)/n(Pu)$	2.192 92(49)	$m(^{240}Pu)/m(Pu)$	2.201 90(49)
$n(^{241}Pu)/n(Pu)$	0.020 55(27)	$m(^{241}Pu)/m(Pu)$	0.020 72(27)
$n(^{242}Pu)/n(Pu)$	0.007 402(76)	$m(^{242}Pu)/m(Pu)$	0.007 495(77)

The molar mass of the uranium in this sample is 237.452 936(98) $g \cdot mol^{-1}$
The molar mass of the plutonium in this sample is 239.074 733 7(86) $g \cdot mol^{-1}$

From the certified values, the following amount contents and mass fractions are derived:

$8.312\,51(76) \cdot 10^{-5}$	mol (U) · g^{-1} (solution)
$3.819\,67(71) \cdot 10^{-3}$	g (^{235}U) · g^{-1} (solution)
$15.861\,6(18) \cdot 10^{-3}$	g (^{238}U) · g^{-1} (solution)
$19.738\,3(18) \cdot 10^{-3}$	g (U) · g^{-1} (solution)
$3.086\,1(13) \cdot 10^{-6}$	mol (Pu) · g^{-1} (solution)
$7.213\,4(30) \cdot 10^{-4}$	g (^{239}Pu) · g^{-1} (solution)
$7.378\,1(31) \cdot 10^{-4}$	g (Pu) · g^{-1} (solution)

NOTES

1. This Spike Isotopic Reference Material is traceable to the SI in the shortest possible way. The values of the U and Pu isotope ratios were measured at IRMM and are traceable to the SI via the values of the isotope ratios of the isotopic reference materials IRMM-183, 184, 185, 186, 187 for uranium and IRMM-290 for plutonium. The U and Pu content of this spike are traceable to the SI via reference materials NBL CRM-116, EC NRM-101 and CETAMA MP2. Measurements calibrated by this Isotopic Reference Material have therefore the potential of being traceable to the SI.

2. All uncertainties indicated in this certificate are expanded uncertainties $U = k \cdot u_c$ where u_c is the combined standard uncertainty estimated following the ISO/BIPM Guide to the Expression of Uncertainty in Measurement. They are given in parentheses and include a coverage factor k=2. They apply to the last two digits of the value.

3. The IRMM-1027m was prepared by metrological weighing of U metals (NBL CRM 116, EC NRM 101) and Pu metal (CETAMA MP2), dissolution in HNO_3, subsequently dispensing by metrological weighing into individual units, drying and conditioning in cellulose acetate butyrate (CAB).

4. IRMM-1027m is delivered in individual glass (penicillin) vials each containing about 50 mg U and 1.8 mg Pu.

5. Values for isotope amount ratios, isotopic compositions and concentrations are valid for 01 November 2009. This certificate is valid until September 2011; the validity may be extended after further tests on the stability of the spike material are carried out.

6. It is recommended to store the vials in vertical position.

7. The half lives used in the calculations are

^{238}Pu : 8.77 (03) $\cdot 10^1$ a[1]
^{239}Pu : 2.411 (03) $\cdot 10^4$ a[1]
^{240}Pu : 6.563 (07) $\cdot 10^3$ a[1]
^{241}Pu : 1.432 5(24) $\cdot 10^1$ a[2]
^{242}Pu : 3.735 (11) $\cdot 10^5$ a[1]
^{244}Pu : 8.00 (09) $\cdot 10^7$ a[1]

8. The atomic masses, used in the calculations, are[3]

^{233}U : 233.039 635 2 (58) g·mol^{-1}
^{234}U : 234.040 952 1 (40) g·mol^{-1}
^{235}U : 235.043 929 9 (40) g·mol^{-1}
^{236}U : 236.045 568 0 (40) g·mol^{-1}
^{238}U : 238.050 788 2 (40) g·mol^{-1}

^{238}Pu : 238.049 559 9 (40) g·mol^{-1}
^{239}Pu : 239.052 163 4 (40) g·mol^{-1}
^{240}Pu : 240.053 813 5 (40) g·mol^{-1}
^{241}Pu : 241.056 851 5 (40) g·mol^{-1}
^{242}Pu : 242.058 742 6 (40) g·mol^{-1}
^{244}Pu : 244.064 204 (10) g·mol^{-1}

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[1] IAEA, Decay data of the Transactinium Nuclides, Technical Reports Series No. 261, 1986
[2] R. Wellum, A. Verbruggen, R. Kessel, J. Anal. At. Spectrom., 2009, 24, 801 - 807
[3] G. Audi and A.H. Wapstra, The 2003 atomic mass evaluation, Nucl Phys A729 (2003) 337-676.

9. The vials should be handled with great care and by experienced personnel in a laboratory environment suitably equipped for the safe handling of radioactive materials.

10. Full details of the certification procedure can be found in the Preparation and Certification Report.[4]

Chemical preparation and ampouling of this IRM were accomplished by J. Bauwens, F. Kehoe and R. Eykens.

The isotopic verification measurements were carried out by F. Kehoe, S. Richter and H. Kühn for uranium and plutonium on samples chemically prepared by R. Jakopic and F. Kehoe. Measurements of isotopic ratios were calibrated against synthetic isotopic mixtures prepared by R. Eykens for uranium and J. Broothaerts for plutonium.

Metrological weighings required in the preparation and certification were performed by F. Kehoe, U. Jacobsson and R. Eykens.

The overall co-ordination leading to the establishment, certification and issuance of this Spike Isotopic Reference Material was performed by A. Verbruggen.

B-2440 GEEL
October 2009

H. Emons
Head
Reference Materials Unit

[4] A. Verbruggen, J. Bauwens, R. Eykens, F. Kehoe, H. Kühn, Y. Kushigeta, U. Jacobsson, R. Jakopic, S. Richter, Y. Aregbe, Preparation and Certification of IRMM-1027m, Large-Sized Dried (LSD) spike, report EUR***** EN

Table 1: list of vial numbers, mass of solution before drying

N°	Mass (g)	N°	Mass (g)	N°	Mass (g)	N°	Mass (g)	N°	Mass (g)	N°	Mass (g)	N°	Mass (g)
1	2.4998	51	2.5183	101	2.5207	151	2.4975	201	2.4995	251	2.5004	301	2.4985
2	2.5064	52	2.5059	102	2.4972	152	2.5084	202	2.5248	252	2.4949	302	2.4929
3	2.5031	53	2.4977	103	2.4989	153	2.4956	203	2.4974	253	2.5061	303	2.4945
4	2.5324	54	2.4950	104	2.5016	154	2.5005	204	2.4966	254	2.4946	304	2.5119
5	2.4978	55	2.5121	105	2.5295	155	2.4995	205	2.5106	255	2.5066	305	2.5043
6	2.5332	56	2.5075	106	2.5027	156	2.5063	206	2.5039	256	2.4967	306	2.4927
7	2.5260	57	2.5236	107	2.5051	157	2.4974	207	2.5210	257	2.5029	307	2.4997
8	2.5107	58	2.4968	108	2.5191	158	2.5237	208	2.5217	258	2.5006	308	2.4936
9	2.5009	59	2.4971	109	2.5020	159	2.4989	209	2.4942	259	2.4981	309	2.5060
10	2.5257	60	2.5090	110	2.4970	160	2.5017	210	2.5054	260	2.5013	310	2.5093
11	2.5216	61	2.5052	111	2.4969	161	2.4957	211	2.5279	261	2.4926	311	2.5097
12	2.5032	62	2.5103	112	2.5132	162	2.4991	212	2.5018	262	2.4990	312	2.5037
13	2.4977	63	2.5023	113	2.4938	163	2.5035	213	2.4983	263	2.4926	313	2.4960
14	2.5176	64	2.4978	114	2.5075	164	2.5232	214	2.4956	264	2.4974	314	2.4946
15	2.4995	65	2.5120	115	2.5015	165	2.4945	215	2.4968	265	2.5106	315	2.5247
16	2.5179	66	2.5162	116	2.5184	166	2.5025	216	2.5198	266	2.4963	316	2.5024
17	2.5197	67	2.5038	117	2.5080	167	2.4962	217	2.4935	267	2.5008	317	2.5183
18	2.4952	68	2.5209	118	2.5179	168	2.4928	218	2.4938	268	2.5157	318	2.5194
19	2.5004	69	2.4962	119	2.4995	169	2.5176	219	2.4999	269	2.5152	319	2.5188
20	2.5061	70	2.4948	120	2.5035	170	2.5216	220	2.5209	270	2.5194	320	2.4941
21	2.5003	71	2.5111	121	2.5003	171	2.5015	221	2.5022	271	2.5039	321	2.5266
22	2.4999	72	2.5074	122	2.5219	172	2.5199	222	2.5194	272	2.5259	322	2.5014
23	2.5017	73	2.4994	123	2.4989	173	2.4931	223	2.4974	273	2.4956	323	2.5192
24	2.5078	74	2.5057	124	2.5013	174	2.4954	224	2.4954	274	2.5099	324	2.5142
25	2.5054	75	2.5244	125	2.4954	175	2.5073	225	2.5196	275	2.5185	325	2.5055
26	2.5195	76	2.5021	126	2.5009	176	2.5247	226	2.5179	276	2.4962	326	2.5161
27	2.5045	77	2.5042	127	2.5108	177	2.5238	227	2.5000	277	2.5233	327	2.4966
28	2.5036	78	2.4963	128	2.5013	178	2.4976	228	2.5060	278	2.5049	328	2.5053
29	2.5244	79	2.5057	129	2.5167	179	2.5128	229	2.4983	279	2.5145	329	2.4966
30	2.4952	80	2.4994	130	2.5015	180	2.5108	230	2.5034	280	2.4956	330	2.5224
31	2.5072	81	2.5088	131	2.5166	181	2.4956	231	2.5224	281	2.5026	331	2.5010
32	2.5306	82	2.5039	132	2.5259	182	2.5003	232	2.4982	282	2.4961	332	2.5010
33	2.4992	83	2.5199	133	2.5218	183	2.4993	233	2.4989	283	2.4994	333	2.4930
34	2.4999	84	2.5032	134	2.5198	184	2.5071	234	2.5055	284	2.5054	334	2.5084
35	2.4985	85	2.4950	135	2.4992	185	2.5026	235	2.5094	285	2.4988	335	2.5195
36	2.5071	86	2.5047	136	2.5242	186	2.5207	236	2.4998	286	2.5176	336	2.5192
37	2.5128	87	2.5001	137	2.5219	187	2.5220	237	2.5254	287	2.5155	337	2.4985
38	2.5117	88	2.5033	138	2.4994	188	2.5050	238	2.5186	288	2.5027	338	2.5059
39	2.5024	89	2.5062	139	2.5058	189	2.5136	239	2.5030	289	2.5171	339	2.5191
40	2.5070	90	2.5026	140	2.4955	190	2.4996	240	2.4974	290	2.5222	340	2.4969
41	2.4953	91	2.5049	141	2.5025	191	2.5227	241	2.5120	291	2.4931	341	2.5229
42	2.5273	92	2.5184	142	2.4947	192	2.5044	242	2.4944	292	2.4993	342	2.5079
43	2.5285	93	2.4971	143	2.5078	193	2.5016	243	2.5052	293	2.4999	343	2.5096
44	2.5212	94	2.5113	144	2.5096	194	2.5207	244	2.5048	294	2.5032	344	2.5276
45	2.5071	95	2.5200	145	2.5085	195	2.5226	245	2.4993	295	2.5031	345	2.5225
46	2.5189	96	2.5039	146	2.5220	196	2.5225	246	2.5064	296	2.5198	346	2.5050
47	2.5272	97	2.4925	147	2.5046	197	2.4992	247	2.5167	297	2.4954	347	2.5153
48	2.4969	98	2.4992	148	2.4952	198	2.5256	248	2.5123	298	2.4950	348	2.4994
49	2.5021	99	2.5121	149	2.5211	199	2.5215	249	2.5002	299	2.5122	349	2.4942
50	2.4965	100	2.5032	150	2.4978	200	2.5026	250	2.5078	300	2.5112	350	2.4928

Table 1: list of vial numbers, mass of solution before drying (continued)

N°	Mass (g)	N°	Mass (g)	N°	Mass (g)	N°	Mass (g)	N°	Mass (g)	N°	Mass (g)	N°	Mass (g)
351	2.5154	401	2.4957	451	2.5283	501	2.5164	551	2.4963	601	2.4934	651	2.5144
352	2.5203	402	2.5128	452	2.5205	502	2.5144	552	2.5001	602	2.4955	652	2.4971
353	2.4993	403	2.4954	453	2.5194	503	2.4994	553	2.4986	603	2.5006	653	2.4949
354	2.5221	404	2.5270	454	2.5006	504	2.5120	554	2.5022	604	2.4955	654	2.4950
355	2.5202	405	2.4939	455	2.4967	505	2.4926	555	2.5140	605	2.5001	655	2.5209
356	2.4945	406	2.5090	456	2.5017	506	2.4977	556	2.4960	606	2.4989	656	2.4945
357	2.5265	407	2.5166	457	2.5227	507	2.5060	557	2.4933	607	2.5124	657	2.4921
358	2.5014	408	2.5227	458	2.5163	508	2.5040	558	2.5263	608	2.4994	658	2.5223
359	2.5200	409	2.5191	459	2.5035	509	2.4977	559	2.4990	609	2.4963	659	2.5008
360	2.5211	410	2.5222	460	2.5201	510	2.4990	560	2.5197	610	2.4965	660	2.4951
361	2.5213	411	2.4994	461	2.4971	511	2.5022	561	2.4944	611	2.5177	661	2.5124
362	2.5193	412	2.5201	462	2.5007	512	2.5112	562	2.4991	612	2.4927	662	2.5165
363	2.5218	413	2.5034	463	2.4982	513	2.5240	563	2.4941	613	2.4941	663	2.5158
364	2.5195	414	2.5176	464	2.5239	514	2.5204	564	2.4997	614	2.5016	664	2.5157
365	2.4970	415	2.4928	465	2.4998	515	2.4990	565	2.5016	615	2.5082	665	2.5178
366	2.5017	416	2.5088	466	2.4972	516	2.4926	566	2.4939	616	2.5128	666	2.4956
367	2.5031	417	2.4968	467	2.4926	517	2.4929	567	2.5013	617	2.5160	667	2.4986
368	2.4979	418	2.5268	468	2.5072	518	2.5117	568	2.4921	618	2.4955	668	2.5136
369	2.5200	419	2.4946	469	2.5064	519	2.5027	569	2.5035	619	2.4984	669	2.5181
370	2.5185	420	2.5043	470	2.5177	520	2.5152	570	2.4976	620	2.5183	670	2.5217
371	2.5231	421	2.4962	471	2.5016	521	2.5202	571	2.5139	621	2.5152	671	2.5123
372	2.5062	422	2.5025	472	2.4940	522	2.5201	572	2.4943	622	2.5178	672	2.4958
373	2.4920	423	2.5184	473	2.5220	523	2.4998	573	2.5205	623	2.5180	673	2.5209
374	2.5008	424	2.5056	474	2.5227	524	2.4991	574	2.4958	624	2.4948	674	2.4991
375	2.5232	425	2.4925	475	2.4967	525	2.4923	575	2.5200	625	2.4958	675	2.4929
376	2.4954	426	2.5000	476	2.4945	526	2.4990	576	2.5167	626	2.5210	676	2.4980
377	2.5227	427	2.5068	477	2.5032	527	2.5033	577	2.4947	627	2.4991	677	2.4957
378	2.5003	428	2.5203	478	2.5230	528	2.5214	578	2.5071	628	2.4946	678	2.4988
379	2.5213	429	2.4956	479	2.4976	529	2.4969	579	2.5144	629	2.4978	679	2.4958
380	2.5199	430	2.5237	480	2.4923	530	2.5009	580	2.4976	630	2.4992	680	2.4963
381	2.5196	431	2.5204	481	2.5038	531	2.4938	581	2.5138	631	2.5168	681	2.5048
382	2.5041	432	2.4987	482	2.5041	532	2.5233	582	2.4951	632	2.5176	682	2.4925
383	2.4921	433	2.5217	483	2.4982	533	2.5239	583	2.4959	633	2.4968	683	2.4925
384	2.5252	434	2.5028	484	2.5008	534	2.4963	584	2.4924	634	2.4954	684	2.5009
385	2.5179	435	2.4939	485	2.5129	535	2.5015	585	2.4933	635	2.5004	685	2.5030
386	2.5235	436	2.4948	486	2.4975	536	2.4977	586	2.5028	636	2.5165	686	2.5187
387	2.4990	437	2.4959	487	2.4976	537	2.5063	587	2.5184	637	2.4994	687	2.4919
388	2.5001	438	2.5006	488	2.4976	538	2.5137	588	2.5175	638	2.5096	688	2.5045
389	2.5221	439	2.5044	489	2.5156	539	2.5207	589	2.5121	639	2.4988	689	2.5121
390	2.5025	440	2.5052	490	2.5010	540	2.5190	590	2.4932	640	2.5016	690	2.5187
391	2.5002	441	2.4956	491	2.5020	541	2.5190	591	2.5025	641	2.4953	691	2.5005
392	2.5149	442	2.4921	492	2.4938	542	2.5033	592	2.5218	642	2.4932	692	2.4944
393	2.5048	443	2.5007	493	2.4959	543	2.5130	593	2.5011	643	2.4962	693	2.5196
394	2.4959	444	2.5108	494	2.5014	544	2.4954	594	2.4987	644	2.4990	694	2.4958
395	2.5021	445	2.4973	495	2.5175	545	2.5235	595	2.5068	645	2.4939	695	2.4943
396	2.5193	446	2.4946	496	2.5190	546	2.5022	596	2.5238	646	2.4948	696	2.5059
397	2.4936	447	2.5065	497	2.4997	547	2.5174	597	2.5168	647	2.5033	697	2.5143
398	2.5168	448	2.4926	498	2.4983	548	2.5038	598	2.5182	648	2.5004	698	2.4967
399	2.5021	449	2.4982	499	2.4934	549	2.4938	599	2.4923	649	2.5120	699	2.5192
400	2.4973	450	2.4977	500	2.5023	550	2.4977	600	2.5036	650	2.4988	700	2.4958

Table 1: list of vial numbers, mass of solution before drying (continued)

N°	Mass (g)	N°	Mass (g)	N°	Mass (g)	N°	Mass (g)	N°	Mass (g)	N°	Mass (g)		Mass (g)
701	2.5157	751	2.4977	801	2.5150	851	2.5144	901	2.5138	951	2.4943	1001	2.5136
702	2.4960	752	2.5153	802	2.5122	852	2.5132	902	2.5137	952	2.4945	1002	2.5113
703	2.4967	753	2.5165	803	2.5203	853	2.5150	903	2.5185	953	2.4920	1003	2.5135
704	2.4932	754	2.4946	804	2.5137	854	2.5136	904	2.4922	954	2.4968	1004	2.5139
705	2.5023	755	2.5151	805	2.5132	855	2.5147	905	2.4952	955	2.5122	1005	2.4928
706	2.4965	756	2.4944	806	2.4921	856	2.4947	906	2.4939	956	2.5140	1006	2.5134
707	2.4934	757	2.4935	807	2.4988	857	2.4962	907	2.5126	957	2.5123	1007	2.5127
708	2.5010	758	2.4937	808	2.5104	858	2.5110	908	2.5118	958	2.5141	1008	2.5132
709	2.4938	759	2.4976	809	2.5150	859	2.4951	909	2.4940	959	2.5156	1009	2.4920
710	2.4954	760	2.4997	810	2.5143	860	2.5143	910	2.5137	960	2.4938	1010	2.4956
711	2.4947	761	2.4922	811	2.4946	861	2.4922	911	2.4949	961	2.5130	1011	2.5126
712	2.5010	762	2.4947	812	2.5132	862	2.5155	912	2.4957	962	2.5140	1012	2.5132
713	2.5160	763	2.4970	813	2.5153	863	2.4941	913	2.5084	963	2.5081	1013	2.5103
714	2.4979	764	2.5126	814	2.5118	864	2.4927	914	2.5119	964	2.4922	1014	2.4931
715	2.4960	765	2.5016	815	2.5142	865	2.5151	915	2.4979	965	2.4947	1015	2.5197
716	2.4951	766	2.5128	816	2.5121	866	2.4955	916	2.5105	966	2.5098	1016	2.4933
717	2.4988	767	2.5067	817	2.4958	867	2.4935	917	2.5126	967	2.5042	1017	2.4965
718	2.4929	768	2.4977	818	2.4959	868	2.4955	918	2.5148	968	2.5048	1018	2.4955
719	2.4930	769	2.4928	819	2.4928	869	2.4925	919	2.4925	969	2.5092	1019	2.4942
720	2.5213	770	2.5004	820	2.5007	870	2.5082	920	2.5124	970	2.5142	1020	2.5116
721	2.4925	771	2.4977	821	2.5109	871	2.4945	921	2.5145	971	2.4927	1021	2.5123
722	2.4969	772	2.5097	822	2.5145	872	2.4944	922	2.4921	972	2.5120	1022	2.4926
723	2.5008	773	2.4959	823	2.5171	873	2.4949	923	2.5119	973	2.4952	1023	2.5127
724	2.5172	774	2.4953	824	2.5128	874	2.5175	924	2.5150	974	2.5147	1024	2.5133
725	2.5176	775	2.5105	825	2.5138	875	2.4968	925	2.5116	975	2.4921	1025	2.4937
726	2.5011	776	2.4973	826	2.5132	876	2.5115	926	2.5144	976	2.5136	1026	2.5149
727	2.4920	777	2.5086	827	2.5152	877	2.5122	927	2.5132	977	2.4972	1027	2.5125
728	2.5009	778	2.5163	828	2.4936	878	2.4951	928	2.5168	978	2.5110	1028	2.4946
729	2.5132	779	2.5121	829	2.4932	879	2.4965	929	2.5132	979	2.5117	1029	2.4928
730	2.4965	780	2.5099	830	2.4944	880	2.5111	930	2.5113	980	2.4947	1030	2.5132
731	2.4964	781	2.5130	831	2.5145	881	2.5111	931	2.5117	981	2.5101	1031	2.4932
732	2.5163	782	2.5107	832	2.5152	882	2.4948	932	2.5155	982	2.5091	1032	2.4927
733	2.4948	783	2.5008	833	2.5124	883	2.5121	933	2.5138	983	2.5081	1033	2.4935
734	2.4977	784	2.5113	834	2.5183	884	2.5139	934	2.5123	984	2.4955	1034	2.5150
735	2.4985	785	2.4922	835	2.4946	885	2.5129	935	2.5143	985	2.5086	1035	2.5145
736	2.4992	786	2.4942	836	2.4935	886	2.5142	936	2.4948	986	2.4932	1036	2.5114
737	2.5158	787	2.5122	837	2.5136	887	2.5051	937	2.4940	987	2.5123	1037	2.5136
738	2.4968	788	2.4956	838	2.5141	888	2.4934	938	2.5109	988	2.4925	1038	2.4936
739	2.4942	789	2.5116	839	2.4922	889	2.5025	939	2.5143	989	2.5111	1039	2.5121
740	2.4947	790	2.4984	840	2.5159	890	2.5146	940	2.5134	990	2.5167	1040	2.5131
741	2.4939	791	2.5120	841	2.5139	891	2.4931	941	2.5132	991	2.5109	1041	2.4933
742	2.4970	792	2.5155	842	2.4939	892	2.4940	942	2.5132	992	2.5098	1042	2.4931
743	2.4945	793	2.4973	843	2.4934	893	2.4936	943	2.5131	993	2.4932	1043	2.5118
744	2.5211	794	2.5115	844	2.5138	894	2.4922	944	2.4935	994	2.4930	1044	2.5132
745	2.4966	795	2.5141	845	2.4946	895	2.4937	945	2.5132	995	2.5175	1045	2.5005
746	2.5154	796	2.4985	846	2.5142	896	2.5134	946	2.5118	996	2.5133	1046	2.5072
747	2.4951	797	2.5077	847	2.5155	897	2.5142	947	2.4940	997	2.4941	1047	2.4923
748	2.4963	798	2.5167	848	2.5148	898	2.5124	948	2.5118	998	2.5121	1048	2.4927
749	2.4999	799	2.4927	849	2.4932	899	2.5127	949	2.4951	999	2.5112	1049	2.4942
750	2.5129	800	2.5170	850	2.4957	900	2.5118	950	2.5059	1000	2.5145	1050	2.5133

Table 1: list of vial numbers, mass of solution before drying (continued)

N°	Mass (g)	N°	Mass (g)	N°	Mass (g)
1051	2.5109	1101	2.5090	1151	2.5105
1052	2.5123	1102	2.5115	1152	2.5023
1053	2.4946	1103	2.5117	1153	2.5026
1054	2.5083	1104	2.5002	1154	2.4970
1055	2.4922	1105	2.5037	1155	2.5109
1056	2.5093	1106	2.4973	1156	2.5141
1057	2.5150	1107	2.4976	1157	2.5125
1058	2.5019	1108	2.5085	1158	2.5128
1059	2.5081	1109	2.4973	1159	2.5096
1060	2.5207	1110	2.5008	1160	2.5088
1061	2.5082	1111	2.5120	1161	2.5060
1062	2.5067	1112	2.4989	1162	2.4922
1063	2.4949	1113	2.5027	1163	2.5057
1064	2.5039	1114	2.4988	1164	2.5076
1065	2.5030	1115	2.5072	1165	2.5086
1066	2.5111	1116	2.5003	1166	2.5089
1067	2.5088	1117	2.5041	1167	2.5126
1068	2.5087	1118	2.5074	1168	2.5083
1069	2.5112	1119	2.5055	1169	2.5090
1070	2.5058	1120	2.5122	1170	2.5035
1071	2.4988	1121	2.4940	1171	2.5144
1072	2.5135	1122	2.4930	1172	2.5069
1073	2.5046	1123	2.5120	1173	2.5077
1074	2.4994	1124	2.4987	1174	2.5059
1075	2.5106	1125	2.5118	1175	2.5116
1076	2.4989	1126	2.4994	1176	2.5049
1077	2.5129	1127	2.5109	1177	2.5080
1078	2.5091	1128	2.5062	1178	2.5059
1079	2.5033	1129	2.5090	1179	2.5032
1080	2.4999	1130	2.5127	1180	2.5130
1081	2.5099	1131	2.4926	1181	2.5065
1082	2.5111	1132	2.5158	1182	2.5092
1083	2.5121	1133	2.5104	1183	2.5065
1084	2.4937	1134	2.5019	1184	2.5021
1085	2.5150	1135	2.5065	1185	2.4936
1086	2.4966	1136	2.4979	1186	2.5114
1087	2.4981	1137	2.5100	1187	2.5046
1088	2.5024	1138	2.5102	1188	2.5092
1089	2.5077	1139	2.4964	1189	2.5085
1090	2.5124	1140	2.4958	1190	2.5069
1091	2.4959	1141	2.5011	1191	2.5078
1092	2.4977	1142	2.5045	1192	2.5005
1093	2.4958	1143	2.5069	1193	2.5080
1094	2.5055	1144	2.5073	1194	2.5126
1095	2.4972	1145	2.5034	1196	2.5093
1096	2.5008	1146	2.5072	1197	2.4953
1097	2.5040	1147	2.5030	1198	2.4973
1098	2.5134	1148	2.5042	1199	2.5119
1099	2.5103	1149	2.4992	1200	2.5091
1100	2.5112	1150	2.5036	1201	2.5079

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Abstract
Large sized dried spikes (LSD) have become a fundamental part of the fissile material control of irradiated nuclear fuel. In the frame of providing these spikes to the nuclear industry, a new set of LSD Spikes for the determination of uranium and plutonium by isotope dilution mass spectrometry in solutions of spent fuel from reprocessing plants has been prepared and certified for uranium and plutonium isotopic contents. The methodology followed was comparable to that of previous batches. The solution, made by dissolution of the starting materials in nitric acid, was dispensed directly into individual penicillin vials. As in previous campaign an automated system was introduced to dispense and weigh the vials.
The new batch of large size dried spikes contains ca. 50 mg of uranium (235U abundance = 19.5%) and ca. 1.8 mg of plutonium (239Pu abundance = 97.8%) in each individual vial, covered with a light layer of organic material (cellulose acetate butyrate) as stabilizer.
The U and Pu amount content was certified based on values from mass metrology of the validated automated system. Verification of the amount contents of the spike was done by IDMS at IRMM. The values measured for the dried covered spikes agreed well with those calculated from the weights of starting materials dissolved and the weights of the final solution.

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